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THE CRYSTAL STRUCTURE OF TWO NEW OXYAMINE SALTS "DOACl & DOABr"

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THE CRYSTAL STRUCTURE OF TWO NEW OXYAMINE SALTS, "DOACl & DOABr".

Charles M. Bock

and

William M. Hodgson, Capt, USAF

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FOREWORD

The synthesis of new propellant ingredients to obtain desirable chemical and physical characteristics is a continuing effort at this laboratory. Molecular structure is fundamental to the nature of any substance and X-ray diffraction analysis provides a rapid and unequivocal method of determining this structure in many cases. Knowing the precise structure of a new compound enables one to predict the feasibility of synthesis of homologs and an estimate of their stability.

This report describes the determination of the crystal and molecular structure of two salts of a new potential propellant material.

Reviewed and approved for publication by:

W.S. ANDERSON, Chief
Chemical & Materials Branch
Propellant Division

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CONFIDENTIAL ABSTRACT

The crystal and molecular structures of two new oxyamine salts, "DOACl and DOABr" have been determined by X-ray diffraction methods. Bond lengths and angles are all normal suggesting that the analogous tris and tetrakis compounds can be made.

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TABLE OF CONTENTS

Section		Page
I	INTRODUCTION	1
II	EXPERIMENTAL	2
III	SOLUTION AND REFINEMENT OF THE STRUCTURES	4
IV	DISCUSSION	10
V	SUMMARY	13
ACKNOWLEDGEMENTS		14
REFERENCES		15
APPENDIX A-1		16
APPENDIX A-2		19
AUTHOR - BIOGRAPHY		24
DISTRIBUTION		25
FORM 1473		30

ILLUSTRATIONS

Figure		Page
1	Stereo Diagram of The Unit Cell of DOACl Showing The Molecular Structure. Single Unbonded Balls in The Diagram Are The Cl Ions	7

TABLES

Table		Page
I	Positional Thermal Parameters for DOABr and COACl	6
II	Important Interatomic Distances, Angles and Their Standard Errors	8
III	Distance and Angles Evolved in Hydrogen Bonding	9
IV	Bond Distances for Some Compounds Similar to DOACl	11
V	Selected Hydrogen Bonding Information	12

SECTION I

INTRODUCTION

C - methylene bis(oxyamine hydrochloride), "DOACL" and methylene bis(oxyamine hydrobromide), "DOABr" were prepared at AFRPL under an in-house exploratory synthesis program. Details of the synthesis will be reported at a later date. Structural studies of the perchlorate salt are now in progress and the results of those stuides will be presented when complete.

SECTION II

EXPERIMENTAL

Clear colorless needle-shaped crystals of DOACL & DOABR were grown by slow evaporation of a water solution.

The crystals were mounted on glass fibers parallel to their needle axis (a_o). X-ray precision photographs established in the crystal symmetry of both crystals as monoclinic. Crystal data are:

	DOABR	DOACl
ao	4.051 Å ±.008	3.941 ±.002
bo	12.25 ±.01	11.885 ±.010
co	12.94 ±.01	12.486 ±.010
β	$90^o 40'$ ± 20'	90.48^o ±.05^o
ρobs	not measured	1.66 g/cm^3
ρX-ray	2.48 g/cm^3	1.77 g/cm^3
Z	4	4
Space group	$P2_1/c$ (C^5_{2h})	Pz_1/c (C^5_{2n})
a_o: b_o: c_o	0.3306:1: 1.056	0.3316:1: 1.051

Lattice parameter measurements on DOABR were made from precission photographs taken with unfiltered molybdenum radiation (λ = 0.71069). Initial intensity data was taken with a Weissenberg camera using the multiple film technique with three films per pack and exposures of 23 and 3 hours and tube power at 40 K.V. and 30 M.A.. Initial intensity data were collected using Ni - filtered Cu radiation (λ = 1.54178). Reflection intensities were visually estimated by comparison with a calibrated intensity scale.

Lattice parameter and final intensity data on DOACL were derived from data taken on an automated Picker FACS-1 diffractometer. The lattice parameter data were obtained with a least squares lattice parameter refine program supplied with the FACS-1 system using twelve reflections ($\lambda = 1.54178$). A LiF monochromator was used for both the intensity data collection and the lattice parameter data.

All unique data were collected to 128° 2θ, however, because of an interruption caused by the full circle, data from two otherwise unique quadrants were used. Absorption corrections were made on the final DOACL data and were found to be appreciable even though the crystal was small (0.047 m.m. diameter X 0.62 m.m. long, $\mu = 92.7 cm^{-1}$). Peak intensity was estimated using the θ - 2θ scanning method over a two degree range. Background was measured for 10 seconds on each side of the peak and a weighing scheme modeled after that suggested by Stout and Jensen was derived. A reflection was considered to be observed if the counts accumulated during the scan were greater than the estimated background.

SECTION III
SOLUTION AND REFINEMENT OF THE STRUCTURES

The structures were solved using the symbolic addition method of Karle and Karle (1). The DOABR structure was solved first by taking advantage of the short a_o axis and solving the structure in its okl projection. A somewhat equivocal E-map yielded Br atom positions which were introduced into the least squares program ORFLS[2]. Several cycles of least squares varying the 2 independent Bromine Y and Z coordinates reduced the conventional agreement factor R to 26 percent. A Fourier calculation using phases derived from the bromide atom coordinates clearly showed the positions of the carbon, oxygen and nitrogen atoms. With the addition of the other non-hydrogen atoms to the least squares refinement and the inclusion of a scale factor and individual isotropic thermal parameters, the residual fell to 12 percent. Unit weights were used throughout this refinement.

Since the crystallographic similarity between DOACl and DOABr was so striking a least squares refinement calculation was made using the okl data for DOACl and the atom coordinates derived from the DOABr structure. The residual stabilized at 12 percent after two cycles varying the same parameters that had been varied in the DOABr two-dimensional refinement.

The X coordinates for the COACl structure were derived by performing a Sigma-two calculation on the three dimensional DOACl data with all normalized structure factors greater than 1.0.

In addition to the okl reflections whose signs were known from the projected structure, the sign of the 131 $|E| = 2.13$ reflection was arbitrarily set positive thus fixing the three-dimensional origin. From the 72 reflections used as a starting set, 101 additional signs were

determined after three iterations of the data. A three-dimensional E map was computed and from this the relative X coordinates were determined.

Heavy atom coordinates derived from photographic data were used to start the refinement of the diffractometer data on DOACl. Several cycles of least squares varying positional and anisotropic least squares thermal parameters using the program ORFLS (2) brought the residual for observed data to 8 percent. Form factors used for heavy atoms were taken from the International Tables (3). The form factors for hydrogen were taken from the work of Stewart, Davidon and Simpson (4). A difference Fourier was completed and all hydrogen atoms were easily located. Also it was noted that the chlorine form factors used were those for neutral atoms producing a ring-shaped area of positive electron density in the difference Fourier (cf Stout & Jensen) (5). Accordingly, the form factors for Cl^- were introduced along with the hydrogen atoms to the next full matrix refinement. Two additional cycles of refinement brought the residual for non zero observed F's to 6.5 percent. Another difference Fourier was computed showing no anomalies and the refinement was terminated. Thermal parameters for the hydrogen atoms were fixed at the values derived for the attached heavy atoms and no attempt was made to refine that value.

Tables of computed and observed structure factors are presented in Appendix A.

Positional and thermal parameters for DOABr and DOACl are presented in Table I.

TABLE I. POSITIONAL THERMAL PARAMETERS FOR DOABr AND DOACl.

Anistropic thermal parameters are of the form Texptl - $(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl$ Error terms as determined from the least squares refinement are enclosed in ( ). Atoms from the DOABr molecule are enclosed in square brackets[ ].

Atom	x	y	z	B	β_{11}	β_{22}	β_{33}	$2\beta_{12}$	$2\beta_{13}$	$2\beta_{23}$
Cl 1	-0.3894(4)	0.4555(1)	0.1390(1)		0.0221(9)	0.0052(1)	0.0039(1)	-0.0000(3)	0.0005(2)	0.0001(1)
[Br 1]	-	0.4555(4)	0.1408(4)	1.0(1)						
Cl 2	-0.4514(4)	0.1280(1)	0.0864(1)		0.0240(9)	0.0047(1)	0.0048(1)	0.0018(3)	0.0007(3)	0.0003(1)
[Br 2]	-	0.1286(4)	0.0890(4)	1.2(0.1)						
O 1	0.1938(10)	0.3445(4)	0.3417(4)		0.0154(25)	0.0048(4)	0.0054(3)	-0.0002(8)	0.0018(7)	0.0004(3)
[O 1]	-	0.3454(28)	0.3422(27)	0.9(7)						
O 2	-0.1332(10)	0.1976(4)	0.4069(4)		0.0156(24)	0.0049(4)	0.0048(3)	-0.0008(8)	-0.0002(7)	0.0005(3)
[O 2]	-	0.2008(33)	0.4109(32)	2.0(9)						
N 1	-0.0497(14)	0.4308(5)	0.3620(5)		0.0252(34)	0.0043(5)	0.0049(4)	-0.0004(10)	-0.0004(10)	-0.0004(4)
[N 1]	-	0.4269(37)	0.3648(35)	1.2(10)						
N 2	0.1112(13)	0.1643(5)	0.4866(5)		0.0225(33)	0.0047(5)	0.0046(4)	-0.0002(10)	0.0008(9)	0.0004(4)
[N 2]	-	0.1643(32)	0.4865(30)	0.6(0.8)						
C	0.0337(17)	0.2420(6)	0.3158(6)		0.0347(42)	0.0056(6)	0.0043(5)	-0.0004(13)	-0.0002(11)	-0.0002(4)
[C]	-	0.2413(43)	0.3123(40)	1.3(1.0)						
H 1	0.853 (17)	0.258 (6)	0.255 (5)							
H 2	0.242 (17)	0.185 (6)	0.286 (5)							
H 3	0.820 (17)	0.441 (6)	0.289 (6)							
H 4	0.078 (17)	0.492 (6)	0.385 (5)							
H 5	0.808 (17)	0.411 (6)	0.429 (5)							
H 6	0.257 (17)	0.106 (6)	0.457 (5)							
H 7	0.223 (17)	0.270 (6)	0.012 (5)							
H 8	-0.22 (18)	0.370 (6)	0.037 (6)							

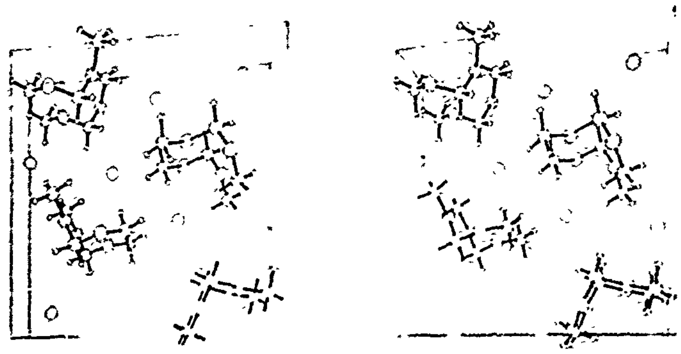

Figure 1. Stereo Diagram of The Unit Cell of DOACl Showing The Molecular Structure. Single Unbonded Balls in The Diagram Are The Cl Ions.

TABLE II. IMPORTANT INTERATOMIC DISTANCES, ANGLES AND THEIR STANDARD ERRORS

Atoms	Distance (Å)	Bond Angles, (deg)
$C-O_1$	1.407(8)	
$C-O_2$	1.418(8)	
$O_1 - N_1$	1.428(6)	
$O_2 - N_2$	1.435(7)	
$Cl_1 - Cl_2$	3.953(4)	
$O_1 - C - O_2$		110.4(5)
$N_1 - O_1 - C$		110.2(4)
$C - H_1$	1.04(5)	
$C - H_2$	1.12(5)	
$H_1 - C - H_2$		110(1)

TABLE III. DISTANCE AND ANGLES EVOLVED IN HYDROGEN BONDING

Atoms	N-Cl(Å)	N-H(Å)	H---Cl(Å)	AN-H. . . Cl(deg)
$N_1 - H_3 .. Cl_1$	3.09	1.05	2.05	173.8 deg
$N_1 - H_4 .. Cl_2$	3.13	0.92	2.23	168.8
$N_1 - H_5 .. Cl_2$	3.13	1.02	2.29	173.4
$N_2 - H_6 .. Cl_1$	3.13	0.98	2.21	157.0
$N_2 - H_7 .. Cl_2$	3.25	0.94	2.31	172.5
$N_2 - H_8 .. CL_1$	3.10	0.88	2.23	173.3

SECTION IV

DISCUSSION

The configuration of the DOACl molecular structure and its relationship to the unit cell chosen, is shown in the stereodiagram Figure I. The stereoplots were made with the program ORTEP (6).

Important interatomic distances and angles and their estimated standard errors were calculated with the program ORFEE (7) and are listed in Tables II and III.

From the inspection of the bond distances and angles about the carbon atom it is apparent that tetrhedral symmetry has been maintained. Further, the DOA molecule has within the limits of error a two-fold axis of symmetry. The difference between the carbon oxygen bonds is 0.011Å ±0.012 Å. The difference between the oxygen-nitrogen bonds is 0.007Å ±0.009Å. The difference between the two carbon-oxygen-nitrogen bond angles is 0.9 ± 0.7 .

TABLE IV. BOND DISTANCES FOR SOME COMPOUNDS SIMILAR TO DOACl

Compound	Distance O-C	Distance O-N	Reference
CH_3ONH_3	1.46 ± 0.04	1.42 ± 0.02	Laurent & Rerat (1964)
CH_3ONH_2	1.44 ± 0.02	1.37 ± 0.02	Brockway, Beach & Pauling (1935)
$(CH_3)_3$ NO HCl		1.425 ± 0.011	Caron & Donohue (1966)
$CH_2O_2(NH_3)_2^{++}Cl_2^{--}$	1.418 ± 0.011	1.431 ± 0.009+	This work

As shown in Table IV, the average carbon-oxygen and oxygen-nitrogen bonds for DOACl are in good agreement with distances determined for similarly bonded molecules, and preliminary work on the structure of the DOA perchlorate salt shows similar molecular symmetry and bond distances.

The separation of the chlorine atoms at 3.95 Å is only slightly greater than one would have predicted on the basis of the usual van der Waals radii of 1.80.

Hydrogen bonding between the amine groups and the chlorine ions seems to be in good agreement with other work (See Table V.).

TABLE V. SELECTED HYDROGEN BONDING INFORMATION

Compound	N...Cl(Å)	N-H(Å)	H...Cl(Å)	AN-H...Cl(deg)	Method of Determination	Reference
Methoxyamire hydrochloride	3.10	-	-	-	X-ray	Laurent & Rerat (1964)
	3.17	-	-	-		
Hydroxylammonium chloride	3.202(6)	1.024(11)	2.256(13)	153.2(9)	Neutron	Padmanabhan(1967)
		1.017(9)	2.248(16)	157.3(8)		
		1.019(9)	2.260(14)	156.2(9)		
DOACL (Averages)	3.14	0.97	2.22	170	X-ray	This work

SECTION V

SUMMARY

In summary, the structure shows unequivocally that the synthesis of the bis oxyamine cation was successful and that there is no reason to believe on stereochemical grounds, that the tris and tetrakis oxyamine radicals cannot also be made.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the substantial help they received from Mr. Stephen Rose in programing many of the required calculations for our computers.

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APPENDIX A-1

DOACL observed and computed structure factors.

H	K	L	FOB	FCA
-4	0	-6	60	57
-4	0	-4	44	49
-4	0	-2	349	324
-4	0	0	222	188
-4	0	2	307	285
-4	0	4	176	189
-4	0	6	235	243
-4	1	-6	95	103
-4	1	-5	139	141
-4	1	-4	107	104
-4	1	-3	277	277
-4	1	-2	103	102
-4	1	-1	124	137
-4	1	0	162	142
-4	1	1	323	294
-4	1	2	68	65
-4	1	3	40	35
-4	1	4	187	180
-4	1	5	60	56
-4	1	6	163	171
-4	1	7	106	106
-4	2	-6	72	67
-4	2	-5	206	201
-4	2	-4	155	156
-4	2	-3	145	151
-4	2	-2	0	1
-4	2	-1	118	135
-4	2	0	114	108
-4	2	1	11	14
-4	2	2	184	173
-4	2	3	32	45
-4	2	4	84	95
-4	2	5	88	84
-4	2	6	161	157
-4	3	-6	15	17
-4	3	-5	8	12
-4	3	-4	0	13
-4	3	-3	44	42
-4	3	-2	48	47
-4	3	-1	111	116
-4	3	0	75	85
-4	3	1	225	216
-4	3	2	202	198
-4	3	3	304	308
-4	3	4	88	96
-4	3	5	36	36
-4	3	6	263	246
-4	4	-5	34	38
-4	4	-4	221	213
-4	4	-3	198	196
-4	4	-2	175	179
-4	4	-1	120	119
-4	4	0	122	127
-4	4	1	183	179
-4	4	2	212	204
-4	4	3	92	94
-4	4	4	52	50
-4	4	5	174	164
-4	5	-4	16	24
-4	5	-3	97	99

H	K	L	FOB	FCA
-4	5	-2	335	337
-4	5	-1	88	96
-4	5	0	32	29
-4	5	1	9	21
-4	5	2	108	103
-4	5	3	107	105
-4	5	4	37	28
-4	6	-3	29	30
-4	6	-2	78	81
-4	6	-1	114	111
-4	6	0	82	74
-4	6	1	246	251
-4	6	2	0	11
-4	6	3	95	89
-3	0	-10	120	121
-3	0	-8	76	84
-3	0	-6	114	125
-3	0	-4	63	76
-3	0	-2	137	132
-3	0	0	206	180
-3	0	2	528	456
-3	0	4	101	106
-3	0	6	272	302
-3	0	8	95	106
-3	0	10	147	154
-3	1	-10	62	62
-3	1	-9	242	257
-3	1	-8	58	70
-3	1	-7	73	81
-3	1	-6	101	112
-3	1	-5	348	389
-3	1	-4	212	214
-3	1	-3	152	156
-3	1	-2	19	7
-3	1	-1	305	298
-3	1	0	318	279
-3	1	1	0	2
-3	1	2	65	66
-3	1	3	136	128
-3	1	4	306	298
-3	1	5	197	210
-3	1	6	181	203
-3	1	7	29	50
-3	1	8	82	94
-3	1	9	309	333
-3	1	10	51	55
-3	1	11	5	12
-3	2	-10	0	3
-3	2	-9	26	29
-3	2	-8	150	155
-3	2	-7	214	235
-3	2	-6	127	143
-3	2	-5	0	6
-3	2	-4	169	178
-3	2	-3	292	308
-3	2	-2	209	216
-3	2	-1	289	281
-3	2	0	239	220
-3	2	1	67	56
-3	2	2	160	150

H	K	L	FOB	FCA
-3	2	3	184	185
-3	2	4	135	129
-3	2	5	112	125
-3	2	6	158	183
-3	2	7	11	24
-3	2	8	62	70
-3	2	9	64	71
-3	2	10	112	118
-3	3	-10	108	109
-3	3	-9	0	20
-3	3	-8	67	72
-3	3	-7	76	77
-3	3	-6	19	26
-3	3	-5	33	34
-3	3	-4	67	83
-3	3	-3	269	281
-3	3	-2	12	13
-3	3	-1	169	179
-3	3	0	366	356
-3	3	1	200	195
-3	3	2	36	35
-3	3	3	201	196
-3	3	4	404	434
-3	3	5	68	80
-3	3	6	29	32
-3	3	7	223	246
-3	3	8	185	202
-3	3	9	100	108
-3	3	10	6	3
-3	4	-10	108	111
-3	4	-9	130	133
-3	4	-8	141	143
-3	4	-7	118	121
-3	4	-6	52	66
-3	4	-5	197	213
-3	4	-4	323	367
-3	4	-3	92	95
-3	4	-2	0	5
-3	4	-1	0	18
-3	4	0	281	284
-3	4	1	251	252
-3	4	2	47	50
-3	4	3	86	96
-3	4	4	97	104
-3	4	5	149	170
-3	4	6	45	54
-3	4	7	194	206
-3	4	8	215	226
-3	4	9	126	117
-3	4	10	67	72
-3	5	-9	73	68
-3	5	-8	286	286
-3	5	-7	108	115
-3	5	-6	257	283
-3	5	-5	130	143
-3	5	-4	152	169
-3	5	-3	0	17
-3	5	-2	81	97
-3	5	-1	66	81
-3	5	0	65	72

H	K	L	FOB	FCA	H	K	L	FOB	FCA	H	K	L	FOB	FCA
-3	5	1	155	171	-3	9	-4	0	3	-2	3	6	119	135
-3	5	2	39	47	-3	9	-3	68	72	-2	3	7	179	197
-3	5	3	110	128	-3	9	-2	57	64	-2	3	8	171	185
-3	5	4	58	59	-3	9	-1	122	127	-2	3	9	159	175
-3	5	5	83	86	-3	9	0	127	133	-2	3	10	69	66
-3	5	6	116	132	-3	9	1	174	180	-2	3	11	92	87
-3	5	7	108	112	-3	9	2	0	2	-2	3	12	19	16
-3	5	8	126	125	-3	9	3	228	236	-2	4	1	139	134
-3	5	9	7	11	-3	9	4	200	198	-2	4	2	185	178
-3	6	-8	7	9	-3	9	5	112	105	-2	4	3	515	517
-3	6	-7	0	12	-3	10	-3	112	105	-2	4	4	97	103
-3	6	-6	74	85	-3	10	-2	140	139	-2	4	5	107	103
-3	6	-5	44	49	-3	10	0	79	70	-2	4	6	201	204
-3	6	-4	125	133	-3	10	1	6	23	-2	4	7	131	133
-3	6	-3	130	149	-3	10	2	160	151	-2	4	8	195	220
-3	6	-2	0	4	-3	10	3	61	64	-2	4	9	97	107
-3	6	-1	193	225	-2	0	-12	186	192	-2	4	10	17	20
-3	6	0	172	178	-2	0	-10	46	53	-2	4	11	101	105
-3	6	1	324	357	-2	0	2	467	747	-2	4	12	166	155
-3	6	2	29	24	-2	0	4	29	36	-2	5	1	360	351
-3	6	3	151	170	-2	0	6	538	522	-2	5	2	148	146
-3	6	4	11	5	-2	0	8	139	149	-2	5	3	180	183
-3	6	5	348	377	-2	0	10	119	132	-2	5	4	146	152
-3	6	6	80	86	-2	0	12	70	73	-2	5	5	129	132
-3	6	7	22	37	-2	1	-13	115	97	-2	5	6	93	109
-3	6	8	0	11	-2	1	-12	17	13	-2	5	7	152	165
-3	6	9	190	177	-2	1	-11	29	43	-2	5	8	51	58
-3	7	-8	81	77	-2	1	-10	89	85	-2	5	9	66	75
-3	7	-7	20	27	-2	1	-7	131	135	-2	5	10	254	258
-3	7	-6	176	179	-2	1	1	71	56	-2	5	11	8	22
-3	7	-5	66	66	-2	1	2	209	169	-2	5	12	126	110
-3	7	-4	156	168	-2	1	3	33	25	-2	6	1	51	52
-3	7	-3	166	187	-2	1	4	209	191	-2	6	2	35	39
-3	7	-2	317	360	-2	1	5	153	148	-2	6	3	489	515
-3	7	-1	0	2	-2	1	6	224	222	-2	6	4	153	157
-3	7	0	92	99	-2	1	7	130	127	-2	6	5	100	99
-3	7	1	201	231	-2	1	8	115	128	-2	6	6	80	86
-3	7	2	108	112	-2	1	9	163	187	-2	6	7	264	290
-3	7	3	77	89	-2	1	10	49	62	-2	6	8	105	106
-3	7	4	148	152	-2	1	11	117	124	-2	6	9	52	56
-3	7	5	139	150	-2	1	12	73	76	-2	6	10	16	25
-3	7	6	60	59	-2	1	13	189	167	-2	6	11	63	54
-3	7	7	138	137	-2	2	0	114	99	-2	7	0	141	153
-3	7	8	81	73	-2	2	1	158	124	-2	7	1	131	132
-3	8	-7	90	86	-2	2	2	332	285	-2	7	2	30	29
-3	8	-6	78	74	-2	2	3	262	235	-2	7	3	223	244
-3	8	-5	8	24	-2	2	4	373	351	-2	7	4	100	115
-3	8	-4	216	223	-2	2	5	307	293	-2	7	5	43	49
-3	8	-3	118	134	-2	2	6	141	130	-2	7	6	225	240
-3	8	-2	57	77	-2	2	7	228	237	-2	7	7	240	252
-3	8	-1	0	14	-2	2	8	315	350	-2	7	8	60	71
-3	8	0	102	114	-2	2	9	146	162	-2	7	9	9	18
-3	8	1	0	2	-2	2	10	50	56	-2	7	10	196	187
-3	8	2	128	134	-2	2	11	248	259	-2	8	1	151	162
-3	8	3	112	120	-2	2	12	153	143	-2	8	2	62	75
-3	8	4	0	13	-2	3	1	242	208	-2	8	3	0	3
-3	8	5	42	42	-2	3	2	147	144	-2	8	4	65	84
-3	8	6	24	25	-2	3	3	215	204	-2	8	5	34	40
-3	8	7	87	83	-2	3	4	348	348	-2	8	6	0	32
-3	9	-5	74	68	-2	3	5	224	216	-2	8	7	127	132

H	K	L	FOB	FCA	H	K	L	FOB	FCA	H	K	L	FOB	FCA
-2	8	8	223	218	-1	2	12	63	63	-1	7	7	18	15
-2	8	9	54	48	-1	2	13	106	96	-1	7	8	107	118
-2	8	10	21	19	-1	2	14	17	23	-1	7	9	90	83
-2	9	1	239	269	-1	3	1	130	169	-1	7	10	243	234
-2	9	2	105	116	-1	3	2	572	562	-1	7	11	63	54
-2	9	3	74	83	-1	3	3	223	213	-1	7	12	29	22
-2	9	4	291	312	-1	3	4	333	309	-1	8	1	0	14
-2	9	5	200	208	-1	3	5	563	565	-1	8	2	239	251
-2	9	6	29	40	-1	3	6	371	369	-1	8	3	245	250
-2	9	7	74	69	-1	3	7	83	75	-1	8	4	0	20
-2	9	8	88	75	-1	3	8	51	56	-1	8	5	90	90
-2	9	9	46	44	-1	3	9	121	126	-1	8	6	225	230
-2	10	1	0	5	-1	3	10	116	113	-1	8	7	131	135
-2	10	2	95	107	-1	3	11	22	32	-1	8	8	161	168
-2	10	3	57	60	-1	3	12	98	95	-1	8	9	231	229
-2	10	4	65	70	-1	3	13	74	65	-1	8	10	55	49
-2	10	5	72	76	-1	4	1	22	25	-1	8	11	123	108
-2	10	6	134	133	-1	4	2	212	201	-1	9	1	252	272
-2	10	7	0	3	-1	4	3	80	79	-1	9	2	185	202
-2	11	1	0	3	-1	4	4	0	7	-1	9	3	119	132
-2	11	2	115	114	-1	4	5	254	255	-1	9	4	39	46
-2	11	3	64	61	-1	4	6	294	292	-1	9	5	187	195
-2	11	4	92	92	-1	4	7	143	155	-1	9	6	0	14
-2	11	5	75	68	-1	4	8	249	248	-1	9	7	10	22
-2	11	6	49	43	-1	4	9	63	68	-1	9	8	64	65
-2	12	1	52	53	-1	4	10	118	131	-1	9	9	33	27
-2	12	2	291	266	-1	4	11	43	42	-1	9	10	46	34
-2	12	3	24	19	-1	4	12	204	199	-1	10	1	0	1
-1	0	2	770	671	-1	4	13	111	102	-1	10	2	221	238
-1	0	4	635	608	-1	5	1	18	6	-1	10	3	141	146
-1	0	6	468	460	-1	5	2	234	239	-1	10	4	169	187
-1	0	8	259	259	-1	5	3	72	86	-1	10	5	207	217
-1	0	10	0	7	-1	5	4	189	187	-1	10	6	159	162
-1	0	12	54	54	-1	5	5	114	121	-1	10	7	0	5
-1	0	14	173	144	-1	5	6	435	432	-1	10	8	148	131
-1	1	1	138	133	-1	5	7	64	74	-1	10	9	161	133
-1	1	2	878	827	-1	5	8	78	84	-1	11	1	33	32
-1	1	3	570	544	-1	5	9	0	3	-1	11	2	0	2
-1	1	4	404	378	-1	5	10	304	325	-1	11	3	54	66
-1	1	5	107	93	-1	5	11	35	34	-1	11	4	166	163
-1	1	6	253	250	-1	5	12	88	82	-1	11	5	37	41
-1	1	7	464	466	-1	5	13	43	34	-1	11	6	24	32
-1	1	8	117	116	-1	6	1	140	129	-1	11	7	196	170
-1	1	9	152	160	-1	6	2	23	21	-1	11	8	130	108
-1	1	10	145	147	-1	6	3	543	548	-1	12	1	0	17
-1	1	11	264	289	-1	6	4	145	138	-1	12	2	122	116
-1	1	12	52	44	-1	6	5	159	166	-1	12	3	27	24
-1	1	13	148	140	-1	6	6	0	13	-1	12	4	162	143
-1	1	14	89	75	-1	6	7	191	192	-1	12	5	31	25
-1	2	1	235	198	-1	6	8	11	17	-1	12	6	91	77
-1	2	2	209	195	-1	6	9	123	118	-1	13	1	25	24
-1	2	3	72	63	-1	6	10	0	0	-1	13	2	131	114
-1	2	4	97	77	-1	6	11	41	40	-1	13	3	39	31
-1	2	5	65	63	-1	6	12	29	23	0	0	2	267	274
-1	2	6	57	54	-1	7	1	424	433	0	0	4	937	57
-1	2	7	32	51	-1	7	2	132	127	0	0	6	48	65
-1	2	8	36	47	-1	7	3	0	13	0	0	8	156	175
-1	2	9	305	321	-1	7	4	55	62	0	0	10	0	8
-1	2	10	30	33	-1	7	5	298	292	0	0	12	118	134
-1	2	11	176	178	-1	7	6	169	176	0	0	14	172	162

APPENDIX A-2

DOABR observed and computed structure factors.

H	K	L	FOB	FCA
0	1	1	16	15
0	1	2	220	210
0	1	3	22	11
0	1	4	388	393
0	1	5	197	201
0	1	6	96	113
0	1	7	342	352
0	1	8	165	164
0	1	9	0	38
0	1	10	0	23
0	1	11	204	231
0	1	12	0	67
0	1	13	0	34
0	1	14	0	11
0	2	0	20	7
0	2	1	172	178
0	2	2	127	129
0	2	3	422	454
0	2	4	429	456
0	2	5	687	702
0	2	6	136	125
0	2	7	220	217
0	2	8	252	247
0	2	9	425	442
0	2	10	230	247
0	2	11	58	60
0	2	12	153	161
0	2	13	245	228
0	2	14	174	146
0	3	1	235	239
0	3	2	612	695
0	3	3	561	603
0	3	4	136	128
0	3	5	304	308
0	3	6	112	123
0	3	7	75	81
0	3	8	143	162
0	3	9	120	134
0	3	10	81	85
0	3	11	60	65
0	3	12	0	5
0	3	13	128	116
0	3	14	57	44
0	4	0	364	378
0	4	1	546	588
0	4	2	382	404
0	4	3	472	475
0	4	4	62	56
0	4	5	239	229
0	4	6	257	251
0	4	7	159	163
0	4	8	88	101
0	4	9	170	171
0	4	10	116	117
0	4	11	41	42
0	4	12	190	171
0	4	13	98	95
0	5	1	532	540
0	5	2	101	89
0	5	3	114	110

H	K	L	FOB	FCA
0	5	4	271	270
0	5	5	279	269
0	5	6	197	197
0	5	7	95	92
0	5	8	369	379
0	5	9	79	84
0	5	10	221	234
0	5	11	111	113
0	5	12	163	148
0	5	13	91	83
0	6	0	116	101
0	6	1	336	344
0	6	2	178	171
0	6	3	389	374
0	6	4	66	51
0	6	5	149	149
0	6	6	0	12
0	6	7	150	149
0	6	8	18	28
0	6	9	80	92
0	6	10	47	54
0	6	11	55	60
0	6	12	94	75
0	6	13	106	83
0	7	1	298	294
0	7	2	0	5
0	7	3	215	212
0	7	4	288	279
0	7	5	252	256
0	7	6	122	129
0	7	7	108	108
0	7	8	207	214
0	7	9	0	10
0	7	10	139	131
0	7	11	123	109
0	7	12	140	120
0	8	0	33	28
0	8	1	247	239
0	8	2	62	77
0	8	3	69	79
0	8	4	84	91
0	8	5	243	257
0	8	6	209	221
0	8	7	0	11
0	8	8	224	219
0	8	9	103	100
0	8	10	160	140
0	8	11	93	73
0	9	1	171	193
0	9	4	198	210
0	9	5	116	129
0	9	6	170	165
0	9	7	36	42
0	9	8	131	127
0	9	9	79	77
0	9	10	0	1
0	9	11	20	14
0	10	0	291	310
0	10	1	117	118
0	10	2	63	74

H	K	L	FOB	FCA
0	10	3	149	166
0	10	4	152	159
0	10	5	38	43
0	10	6	186	194
0	10	7	77	75
0	10	8	102	99
0	10	9	175	159
0	10	10	121	103
0	11	1	45	41
0	11	2	168	174
0	11	3	138	137
0	11	4	32	31
0	11	5	84	77
0	11	6	0	10
0	11	7	232	207
0	11	8	60	48
0	12	0	206	204
0	12	1	61	72
0	12	2	55	55
0	12	3	34	33
0	12	4	228	211
0	12	5	82	72
0	12	6	182	148
0	12	7	5	9
0	13	1	59	54
0	13	2	171	148
0	13	3	179	162
0	13	4	53	54
0	13	5	50	40
1	0	0	124	90
1	0	2	452	418
1	0	4	174	172
1	0	6	205	195
1	0	8	202	207
1	0	10	76	90
1	0	12	128	129
1	0	14	106	100
1	1	0	80	59
1	1	1	205	194
1	1	2	917	930
1	1	3	589	584
1	1	4	104	105
1	1	5	242	215
1	1	6	417	421
1	1	7	419	419
1	1	8	94	99
1	1	9	256	264
1	1	10	33	52
1	1	11	315	331
1	1	12	42	49
1	1	13	102	103
1	1	14	124	101
1	2	0	682	607
1	2	1	224	233
1	2	2	307	296
1	2	3	700	708
1	2	4	260	255
1	2	5	172	179
1	2	6	316	322
1	2	7	288	290

H	K	L	FOB	FCA
1	2	8	61	53
1	2	9	149	155
1	2	10	85	93
1	2	11	0	7
1	2	12	60	58
1	2	13	76	71
1	2	14	66	55
1	3	0	319	309
1	3	1	299	295
1	3	2	496	489
1	3	3	421	405
1	3	4	53	51
1	3	5	27	21
1	3	6	216	211
1	3	7	89	89
1	3	8	92	91
1	3	9	82	85
1	3	10	0	8
1	3	11	148	147
1	3	12	170	165
1	3	13	120	107
1	4	0	279	280
1	4	1	85	79
1	4	2	52	52
1	4	3	10	14
1	4	4	214	218
1	4	5	97	91
1	4	6	402	406
1	4	7	149	156
1	4	8	159	161
1	4	9	143	158
1	4	10	260	268
1	4	11	154	151
1	4	12	116	110
1	4	13	91	78
1	5	0	421	414
1	5	1	83	85
1	5	2	53	43
1	5	3	71	79
1	5	4	529	528
1	5	5	122	112
1	5	6	105	91
1	5	7	125	117
1	5	8	378	400
1	5	9	44	40
1	5	10	0	2
1	5	11	37	38
1	5	12	191	173
1	5	13	71	57
1	6	0	5	13
1	6	1	630	630
1	6	2	243	249
1	6	3	337	343
1	6	4	21	21
1	6	5	266	265
1	6	6	11	29
1	6	7	133	131
1	6	8	131	129
1	6	9	30	42
1	6	10	0	15

H	K	L	FOB	FCA
1	6	11	89	84
1	6	12	0	3
1	7	0	78	79
1	7	1	86	83
1	7	2	37	53
1	7	3	100	94
1	7	4	247	252
1	7	5	11	12
1	7	6	134	128
1	7	7	0	15
1	7	8	245	259
1	7	9	0	7
1	7	10	117	109
1	7	11	65	51
1	7	12	182	152
1	8	0	200	201
1	8	1	109	118
1	8	2	125	119
1	8	3	174	177
1	8	4	184	193
1	8	5	99	106
1	8	6	270	280
1	8	7	134	136
1	8	8	72	62
1	8	9	99	93
1	8	10	231	202
1	8	11	100	84
1	9	0	0	9
1	9	1	119	124
1	9	2	191	191
1	9	3	309	326
1	9	4	0	1
1	9	5	133	131
1	9	6	36	46
1	9	7	78	78
1	9	8	0	3
1	9	9	93	86
1	9	10	93	80
1	10	0	103	111
1	10	1	0	9
1	10	2	185	194
1	10	3	139	153
1	10	4	131	141
1	10	5	122	128
1	10	6	35	41
1	10	7	168	152
1	10	8	27	22
1	10	9	195	158
1	11	0	120	120
1	11	1	70	81
1	11	2	53	61
1	11	3	83	87
1	11	4	91	90
1	11	5	157	158
1	11	6	0	1
1	11	7	210	186
1	11	8	143	119
1	12	0	311	307
1	12	1	45	48
1	12	2	36	36

H	K	L	FOB	FCA
1	12	3	93	82
1	12	4	167	157
1	12	5	28	25
1	12	6	77	62
1	13	0	127	116
1	13	1	81	76
1	13	2	188	159
1	13	3	27	21
2	0	0	581	405
2	0	2	577	497
2	0	4	413	400
2	0	6	193	195
2	0	8	105	104
2	1	0	385	290
2	1	1	429	363
2	1	2	302	278
2	1	3	65	67
2	1	4	171	160
2	1	5	511	512
2	1	6	102	117
2	1	8	67	82
2	1	9	245	269
2	2	1	229	194
2	2	2	293	261
2	2	3	225	206
2	2	4	113	114
2	2	5	128	128
2	2	6	167	165
2	2	8	130	138
2	2	9	197	214
2	2	10	178	190
2	2	11	122	125
2	2	12	101	99
2	3	0	431	374
2	3	1	11	2
2	3	2	276	264
2	3	3	79	78
2	3	4	184	177
2	3	5	158	152
2	3	8	0	14
2	3	9	37	44
2	3	10	136	142
2	3	11	52	53
2	3	12	64	62
2	4	0	128	117
2	4	1	488	452
2	4	2	248	237
2	4	3	339	338
2	4	4	192	190
2	4	5	393	410
2	4	7	164	175
2	4	8	159	181
2	4	9	65	72
2	4	10	219	220
2	4	11	154	149
2	4	12	6	13
2	5	0	37	44
2	5	1	0	26
2	5	2	439	430
2	5	3	305	306

H	K	L	FOB	FCA
2	5	4	188	194
2	5	6	123	129
2	5	7	78	85
2	5	8	158	164
2	5	9	52	60
2	5	10	21	30
2	5	11	80	77
2	6	0	145	137
2	6	1	415	425
2	6	2	163	182
2	6	5	107	123
2	6	6	115	126
2	6	7	35	43
2	6	8	126	126
2	6	9	94	95
2	6	10	118	102
2	6	11	169	155
2	7	2	41	52
2	7	3	138	151
2	7	4	235	257
2	7	5	164	178
2	7	6	200	216
2	7	7	40	52
2	7	8	272	271
2	7	9	76	79
2	7	10	126	108
2	8	0	137	158
2	8	1	75	88
2	8	2	170	191
2	8	3	53	61
2	8	4	144	161
2	8	5	0	3
2	8	6	258	278
2	8	7	122	125
2	8	8	0	11
2	8	9	48	42
2	8	10	218	190
2	9	0	309	343
2	9	1	18	30
2	9	2	172	190
2	9	3	94	106
2	9	4	120	130
2	9	5	65	62
2	9	6	115	110
2	9	7	0	5
2	9	8	0	1
2	9	9	48	39
2	10	0	59	63
2	10	1	163	178
2	10	2	86	89
2	10	3	196	202
2	10	4	17	23
2	10	5	0	14
2	10	6	115	109
2	10	7	143	132
2	11	0	35	36
2	11	1	114	113
2	11	2	66	64
2	11	3	15	29
2	11	4	7	20

H	K	L	FOB	FCA
2	11	5	250	223
2	11	6	49	46
2	12	0	112	109
2	12	1	0	5
2	12	2	192	171
2	12	3	27	27

H	K	L	FOB	FCA

H	K	L	FOB	FCA
0	0	2	96	72
0	0	4	308	389
0	0	6	23	22
0	0	8	100	86
0	0	10	14	4
0	0	12	78	79
0	0	14	107	103
0	0	16	64	69
0	1	1	10	0
0	1	2	87	80
0	1	3	110	99
0	1	4	150	116
0	1	5	118	86
0	1	6	82	68
0	1	7	224	214
0	1	8	93	93
0	1	9	25	29
0	1	10	28	30
0	1	11	191	172
0	1	12	0	10
0	1	13	0	6
0	1	14	29	42
0	1	15	91	88
0	1	16	34	33
0	2	0	103	97
0	2	1	18	17
0	2	2	66	52
0	2	3	195	172
0	2	4	181	160
0	2	5	247	236
0	2	6	102	86
0	2	7	130	115
0	2	8	123	115
0	2	9	209	231
0	2	10	131	121
0	2	11	31	29
0	2	12	72	61
0	2	13	190	148
0	2	14	122	85
0	2	15	39	43
0	2	16	0	5
0	3	1	174	183
0	3	2	233	286
0	3	3	210	203
0	3	4	56	52
0	3	5	186	158
0	3	6	111	96
0	3	7	41	35
0	3	8	39	43
0	3	9	0	14
0	3	10	29	23
0	3	11	0	9
0	3	12	34	24
0	3	13	98	76
0	3	14	55	47
0	3	15	0	3
0	3	16	59	72
0	4	0	144	137
0	4	1	203	186
0	4	2	144	124

H	K	L	FOB	FCA
0	4	3	161	153
0	4	4	43	42
0	4	5	152	130
0	4	6	185	168
0	4	7	55	43
0	4	8	62	52
0	4	9	127	109
0	4	10	131	119
0	4	11	0	11
0	4	12	126	117
0	4	13	90	65
0	4	14	28	24
0	4	15	64	60
0	4	16	44	70
0	5	1	180	180
0	5	2	59	50
0	5	3	56	47
0	5	4	175	165
0	5	5	155	124
0	5	6	114	98
0	5	7	46	40
0	5	8	214	226
0	5	9	35	36
0	5	10	148	125
0	5	11	68	59
0	5	12	166	128
0	5	13	38	42
0	5	14	62	51
0	5	15	0	9
0	6	0	29	27
0	6	1	197	202
0	6	2	53	48
0	6	3	209	223
0	6	4	11	22
0	6	5	105	97
0	6	6	0	12
0	6	7	106	106
0	6	8	0	4
0	6	9	31	27
0	6	10	33	21
0	6	11	42	38
0	6	12	30	40
0	6	13	15	19
0	6	14	0	4
0	6	15	80	103
0	7	1	141	156
0	7	2	37	46
0	7	3	85	92
0	7	4	134	144
0	7	5	140	134
0	7	6	77	83
0	7	7	44	45
0	7	8	148	143
0	7	9	16	6
0	7	10	102	88
0	7	11	73	58
0	7	12	105	101
0	7	13	60	58
0	7	14	65	78
0	8	0	22	27

H	K	L	FOB	FCA
0	8	1	92	103
0	8	2	57	58
0	8	3	25	33
0	8	4	0	12
0	8	5	121	128
0	8	6	171	156
0	8	7	0	9
0	8	8	105	98
0	8	9	117	86
0	8	10	150	129
0	8	11	59	40
0	8	12	97	94
0	8	13	65	63
0	8	14	40	54
0	9	1	106	129
0	9	2	149	187
0	9	3	45	59
0	9	4	75	81
0	9	5	79	79
0	9	6	94	97
0	9	7	53	49
0	9	8	87	56
0	9	9	0	25
0	9	10	0	9
0	9	11	14	17
0	9	12	0	22
0	9	13	43	50
0	10	0	150	172
0	10	1	49	51
0	10	2	30	22
0	10	3	96	101
0	10	4	109	116
0	10	5	53	53
0	10	6	69	74
0	10	7	85	69
0	10	8	63	62
0	10	9	126	116
0	10	10	73	63
0	10	11	0	9
0	10	12	0	8
0	11	1	0	14
0	11	2	84	81
0	11	3	96	98
0	11	4	0	15
0	11	5	57	54
0	11	6	0	16
0	11	7	163	152
0	11	8	0	10
0	11	9	34	34
0	11	10	25	14
0	11	11	112	138
0	12	0	153	176
0	12	1	0	16
0	12	2	34	24
0	12	3	0	3
0	12	4	148	152
0	12	5	32	41
0	12	6	68	65
0	12	7	0	9
0	12	8	27	32

H	K	L	FOB	FCA	H	K	L	FOB	FCA	H	K	L	FOB	FCA
0	12	9	44	45										
0	12	10	0	6										
0	13	1	0	26										
0	13	2	117	115										
0	13	3	86	90										
0	13	4	37	36										
0	13	5	0	2										
0	13	6	33	38										
0	13	7	109	106										
0	13	8	20	29										
0	13	9	0	25										
0	14	0	28	28										
0	14	1	0	2										
0	14	2	0	22										
0	14	3	54	57										
0	14	4	0	23										
0	14	5	59	69										
0	14	6	53	54										
0	14	7	40	50										
0	15	1	71	89										
0	15	2	92	104										
0	15	3	29	36										
0	15	4	0	3										
0	15	5	27	54										

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13. ABSTRACT

The crystal and molecular structures of two new oxyamine salts, "DOACl and DOABr" have been determined by x-ray diffraction methods. Bond lengths and angles are all normal suggesting that the analogous tris and tetrakis compounds can be made.

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